National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1619b

Sulfur in Residual Fuel Oil (0.7 %)

This Standard Reference Material (SRM) is intended for use in the calibration of instruments and the evaluation of methods used in the determination of total sulfur and mercury in fuel oils or materials of similar matrix. A unit of SRM 1619b consists of 100 mL of commercial "No. 6" residual fuel oil as defined by ASTM D396-95 Standard Specification for Fuel Oils [1].

Table 1. Certified Values (Mass Fraction)

Sulfur:	0.6960 %	±	0.0077 %
Mercury:	3.46 ng/kg	±	0.74 ng/kg

Certified Values: The certified sulfur and mercury values are provided in Table 1. The certified sulfur content is based on analyses by isotope dilution thermal ionization mass spectrometry (ID-TIMS) [2]. The certified mercury content is based in cold vapor isotope dilution inductively coupled plasma mass spectrometry (CV-ID-ICP-MS) [3]. Homogeneity testing was performed using X-ray fluorescence (XRF) spectrometry. The uncertainty in the certified value is expressed as an expanded uncertainty and is calculated according to the method in the ISO and NIST Guides [4]. The expanded uncertainty is based on a 95 % prediction interval [5].

Expiration of Certification: The certification of this SRM is valid until **01 July 2008**, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use" section). However, the certification will be nullified if the SRM is damaged, contaminated, or modified.

Stability: This material is considered stable during the period of certification. NIST will monitor this material and will report any significant changes in certification to the purchaser. Registration (see attached sheet) will facilitate notification.

INSTRUCTIONS FOR USE

Because of the viscosity of SRM 1619b, it is recommended that the SRM unit be warmed slowly to between 40 °C and 60 °C and then shaken, or stirred with a clean stirrer, before sampling. Care must be exercised to not introduce entrapped air, which could affect gravimetric measurements and XRF responses. A detailed study to determine if the sulfur components of SRM 1619b will segregate has not been performed at this time.

The SRM bottle should only be opened for the minimum time required to dispense the material. To relate analytical determinations to the certified value in this Certificate of Analysis, a minimum sample mass of 140 mg should be used. After use, the bottle should be tightly recapped and stored under normal laboratory conditions away from direct sunlight.

The overall direction and coordination of the technical measurements leading to certification of this SRM were performed by J.D. Fassett and G.C. Turk of the NIST Analytical Chemistry Division.

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Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 16 May 2006
See Certificate Revision History on Last Page

Robert L. Watters, Jr., Chief
Measurement Services Division

Analytical measurements were performed by W.R. Kelly, R.D. Vocke, Jr., S.E. Long, A.F. Marlow, J.R. Sieber, and J.L. Mann of the NIST Analytical Chemistry Division.

Statistical consultation for this SRM was provided by K.R. Eberhardt of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

SUPPLEMENTAL INFORMATION

Additional properties of SRM 1619b are listed below. These properties were determined by a commercial firm under contract to NIST using ASTM methods. The results are **NOT** certified and are provided only as additional information on the matrix.

Physical Property Test	**ASTM Standard Used**	**Result**
Density @ 15 °C	D1250-80 (1990)	1010.1 kg/m^3
@ 60 °F	D287-92 (1995)	8.5 °API
Flash Point, PMCC	D93-94	93 °C
Pour Point	D97-93	0 °C
Heat of Combustion, Gross	D240-92$^{\epsilon 1}$	41.74 MJ/kg (17947 Btu/lb)
Kinematic Viscosity @ 40 °C	D445-94$^{\epsilon 1}$	322.9 × 10^{-6} m^2/s (322.9 cSt)
@ 50 °C	D445-94$^{\epsilon 1}$	151.1 × 10^{-6} m^2/s (151.1 cSt)
@ 100 °C	D445-94$^{\epsilon 1}$	16.17 × 10^{-6} m^2/s (16.17 cSt)
Carbon	D5291-92	88.0 %
Hydrogen	D5291-92	10.0 %

ASTM Standard Test Methods

D93-94	Standard Test Methods for Flash Point by Pensky-Martens Closed Tester
D97-93	Standard Test Methods for Pour Point of Petroleum Products
D240-92$^{\epsilon 1}$	Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter
D287-92 (1995)	Standard Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)
D445-94$^{\epsilon 1}$	Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)
D1250-80 (1990)$^{\epsilon 1}$	Standard Guide for Petroleum Measurement Tables
D5291-92	Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Petroleum Products and Lubricants

$^{\epsilon 1}$ Indicates that only editorial changes were made to the previous issuance of the ASTM standard.

REFERENCES

[1] ASTM D396-95, *Standard Specification for Fuel Oils*; Annu. Book ASTM Stand., Vol. 05.01, West Conshohocken, PA.

[2] Kelly, W.R.; Paulsen, P.J.; Murphy, K.E.; Vocke, R.D.; Jr.; Chen, L.-T.; *Determination of Sulfur in Fossil Fuels by Isotope Dilution Thermal Ionization Mass Spectrometry*; Anal. Chem., Vol. 66, pp. 2505-2513 (1994).

[3] Christopher, S.J.; Long, S.E.; Rearick, M.S.; *Development of High Accuracy Vapor Generation ICP-MS and Its Application to the Certification of Mercury in Standard Reference Materials*; Anal. Chem., Vol. 73, pp. 2190–2199 (2001).

[4] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, lst ed. International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at http://physics.nist.gov/Pubs/.

[4] Hahn, G.J.; Meeker, W.Q.; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc.: NY (1991).

Certificate Revision Date History: 16 May 2006 (Editorial changes); 01 March 2006 (Editorial changes); 24 January 2006 (This revision reflects an editorial change); 29 November 2004 (This revision reflects the addition of a certified mercury value); 27 July 1998 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.